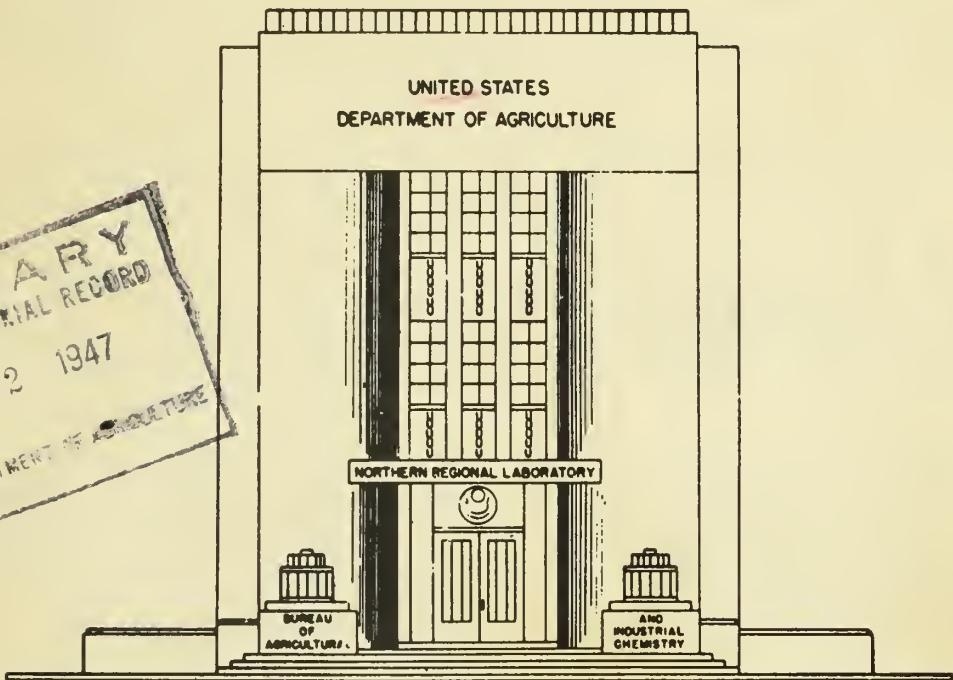


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SOLVENT EXTRACTION OF OILSEEDS



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OILSEED PROCESSING

The processing of oilseeds has been a vital industry for thousands of years. During this time, both the equipment and the methods employed for conducting the various operations have undergone practically continuous evolution from primitive toward modern machinery and practices. The present discussion is limited, however, to developments within the past 50 years and deals chiefly with the present trend toward the use of solvent extraction for processing those oilseeds which, in the past, have been treated by other means.

Many processes are used to recover oil from seeds, but the three common procedures are hydraulic pressing, expeller pressing, and solvent extraction. All three are used widely, frequently in combination.

Hydraulic presses are made in a variety of sizes and types, of which three styles are employed on a large scale in this country. Examples of these include the cage presses found in castor-bean mills, box presses for crushing cottonseed, and plate presses designed for processing linseed. The last two are often referred to as Anglo-American presses.

For processing some kinds of seeds, particularly soybeans, continuous presses such as the Anderson expeller and the French screw press have replaced the hydraulic equipment originally utilized. They have proved quite successful because of lower operating costs per unit of material processed and because they require very little hand labor. Expellers are used throughout the world on a wide variety of oleaginous materials, but in some countries their chief function is to forepress seeds containing a high percentage of oil, that is, to reduce the oil content of the seeds sufficiently to permit the use of solvent extraction for "finishing" the process.

The advantages of using solvents to extract oil from seeds have long been recognized, for extraction ordinarily permits the attaining of a much lower residual content of oil in the meal than does pressing. Many seeds, however, are difficult to treat with solvents because of their physical structure or other characteristics. In addition, extraction is a more complicated process than pressing and has required much more developmental work on the design of suitable equipment in order to make it a practical method of operation. Only in very recent years have some of the most serious obstacles to its use been overcome, and in some applications there still remain many problems to be solved.

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From the standpoint of oil recovery, solvent extraction is the most efficient method known for processing oilseeds. The content of residual oil in the marc seldom exceeds 1 percent, and in the case of soybeans a figure of 0.6 percent is typical, representing an overall recovery of about 96 percent of the oil originally in the beans. Expeller pressing of soybeans yields a meal containing 4 to 5 percent oil, on the basis of 12 percent moisture. To obtain such a product the press recovers, approximately, only 80 percent of the oil. Since the oil commands five times the price of the meal, one striking advantage of extraction is immediately apparent. Hydraulic pressing similarly produces a meal containing 5 to 6 percent oil which, in the case of soybeans, represents an oil recovery of only 75 percent.

Although solvent extraction affords a comparatively high yield of oil, which is the principal advantage of the process, it suffers a number of disadvantages which have caused oil millers to proceed slowly in its adoption. The soybean industry has led all others in the trend toward extraction, but this development is due at least in part to the fact that soybeans are ideally suited to the solvent method of processing. Nearly all other seeds contain higher percentages of oil and consequently are difficult to form into thin, uniform flakes which can be handled in the ordinary types of contacting apparatus. Furthermore, seeds containing a large amount of oil cannot be formed readily into sufficiently rugged flakes, or into any other shape which lends itself to rapid extraction of the oil, without disintegrating. It is for this reason that many operators forepress these raw materials which contain high percentages of oil.

Another disadvantage of the solvent-extraction process is its initially higher cost, when compared with an equivalent expeller mill. Comparisons between the two systems in this respect yield different results, depending on the make and size of equipment under consideration. A prewar estimate indicated that extraction facilities cost one-third more than equivalent expeller equipment, and the fixed charges on the invested capital are therefore somewhat higher in the case of extraction. Other factors which contribute to the overall cost of operation compensate to some extent for the greater fixed charges, and the added revenue which results from the higher yield of oil obtained by extraction much more than justifies any slight disadvantage which the solvent method might have with respect to operating costs.

Solvent extraction is more hazardous than pressing, and in order to conduct the operation safely, it is necessary to employ more highly skilled labor and to maintain a staff of technically trained operators. In many cases, however, the extra cost of such workers is reflected in enhanced value of the products and more efficient operation than would be obtained from less highly qualified operators.

THEORY OF EXTRACTION

The theory of diffusion has been variously applied for correlating data on rates of extraction, predicting extraction time, and designing contacting apparatus. No detailed review of such research will be presented here, but attention should be called to two recent publications^{2,3} from the University of Michigan in which it has been shown that the theory of diffusion can be applied successfully to the extraction of oil from soybean flakes if it is assumed that part of the oil is readily extracted and that the remainder can be leached out only with considerably greater difficulty. This conclusion, arrived at through quantitative measurements, is in

² King, Charles O., Katz, Donald L., and Brier, John C. The solvent extraction of soybean flakes. *Trans. Amer. Inst. Chem. Engin.* 40:533-556. 1944.

³ Osburn, James O., and Katz, Donald L. Structure as a variable in the application of diffusion theory to extraction. *Trans. Amer. Inst. Chem. Engin.* 40:511-531. 1944.

agreement with qualitative observations made by many operators of extraction equipment that the oil removed initially is of higher quality than the smaller proportion which is extracted with greater difficulty in the final stages of the contacting operation. The differences in rate of extraction are attributed largely to non-uniformity in the structure of the solid phase. The observed differences in the quality of the earlier and later fractions of extracted oil, however, point to the character of the solute as having at least some influence upon the deviations from ideal extraction.

The designing and developing of methods for operating extraction apparatus seem to have progressed, by rule-of-thumb methods, considerably faster than has the application of the laws of diffusion and extraction for determining the proper size of apparatus and optimum operating conditions. Research on the theoretical aspects of extraction has made rapid strides within the past few years, however, and promises to contribute greatly toward improving the equipment available in the future.

TYPES OF EXTRACTORS

The simplest form of extractor is a single, stationary kettle having a perforated false bottom. The solids are introduced through an opening in the top, the kettle is closed, and solvent either is pumped through the mass or flows through it by gravity. This type is outmoded and used only on a very small scale or in plants that are nearly obsolete. A variation, however, is the rotary extractor used in this country for processing castor pomace and, to a less extent, other oleaginous materials. It overcomes the chief disadvantage of the single stationary pot, namely, the tendency of the solvent to channel through the solid mass and thereby to extract non-uniformly.

With single extractors of the batch type it is impossible to utilize the principle of countercurrent flow between the solvent and the solids in order to achieve maximum efficiency and to obtain a concentrated miscella. The original method of attaining countercurrent contact is still widely used, mainly in Europe, and consists in the use of several batch extractors in series. Figure 1 is a drawing of an extraction kettle of the type used in Germany. In a typical installation there are 10 of these individual kettles, and solvent is pumped through five or six of them in series. Every few minutes, a completely extracted container is removed from the line, and a freshly filled one is added, the sequence of operations being such as to provide countercurrent, multiple-contact extraction. At any given time, five units are undergoing extraction, one is standing to allow drainage of the solvent from the spent meal, two are being blown with steam to evaporate the entrained solvent, one is being emptied, and one is being filled. The chief use in the United States of batteries of extractors such as these appears to be for the recovery of turpentine, rosin, and other naval stores from waste pine wood.

We are indebted to the German oilseed industry for much of our present-day technology in the field of extraction. After World War I, several methods were developed in that country for providing countercurrent contact continuously between oilseeds and a solvent. Some of these were introduced into the United States, and these were later copied in modified form by American fabricators of equipment. Other manufacturers in this country have recently developed continuous extractors of their own design.

One of the early continuous extractors was that of Hildebrandt, which is widely used in Europe and in three American mills. It is shown in Figure 2 and consists of a vertical U-tube through which the solids are propelled countercurrently to the

solvent by means of slowly revolving screw conveyors which are perforated. One other German system has been imported into the United States, namely, that of Bollmann, and is in use in two American plants. As shown in Figure 3, it consists of a series of sieve-bottomed baskets hanging on chains which revolve somewhat in the manner of a bucket elevator while solvent is sprayed upon the contents. Domestic manufacturers have also adopted this general design, and a large number of American-built plants of this type are now used in the soybean industry.

Figure 4 shows the Bonotto extractor, which is generally similar to the apparatus made by the Allis-Chalmers Manufacturing Company, the V. D. Anderson Company, the Production Engineering Company, and others and which is used on a large scale to process soybeans. The flaked seeds fall by gravity from one plate to another in a vertical column, being propelled around each circular plate in turn by means of a scraper arm before falling through an opening to the next lower shelf. Solvent is introduced at the bottom and flows upward, dissolving oil from the flakes as it passes countercurrently to them, and the miscella overflows at the top.

An extractor which bears a slight resemblance to the Allis-Chalmers model is that designed by Tyca and shown in Figure 5. The seeds pass downward through the vertical column in the same manner as in the Allis-Chalmers equipment, but each shelf has a perforated false bottom. Solvent is sprayed on the material as it passes over each shelf, and the resulting miscella is collected below the false bottom and pumped to higher trays where it is again sprayed on the downward-moving seeds. Figure 6 is a photograph of the remains of the only installation ever made, namely, one in Harburg, Germany.

The Kennedy extractor has been employed for the recovery of cocoa butter, and a small installation for experimental use has recently been installed at the Northern Regional Research Laboratory, Peoria, Ill. As shown in Figure 7, it is a horizontal trough containing a series of paddle wheels, each of which revolves in a close-fitting depression in the floor of the apparatus. Solids are propelled from one depression to the next by the paddle wheels, and solvent passes countercurrently through small holes in the paddles.

Figure 8 is a drawing of the Fauth system of extraction, which is used in two European mills. Countercurrent contact between the solids and the solvent is provided in a series of horizontal screw conveyors, Nos. 4, 6, and 8 in the drawing, following each of which there is a vapor-tight expeller to force most of the entrained solvent out of the marc.

Another type of extractor, used both in Europe and in South America, is the MIAG. As shown in Figure 9, it is a single paddle wheel with many perforated paddles which revolve in an annular race. The seeds are propelled once around the race by means of paddles while the solvent flows in the opposite direction. It is built in comparatively small-sized units which are widely used for the processing of sunflower seeds. A photograph of one of these extractors is shown in Figure 10.

The Ford system of extraction employs an inclined screw conveyor through which the solids and solvent flow countercurrently. The Detrex extractor, shown in Figure 11, is designed for use with trichloroethylene as a solvent and utilizes a horizontal conveyor, as also does a similar process developed by the Engineering Experiment Station of Iowa State College. These three systems were all developed for application on a small scale.

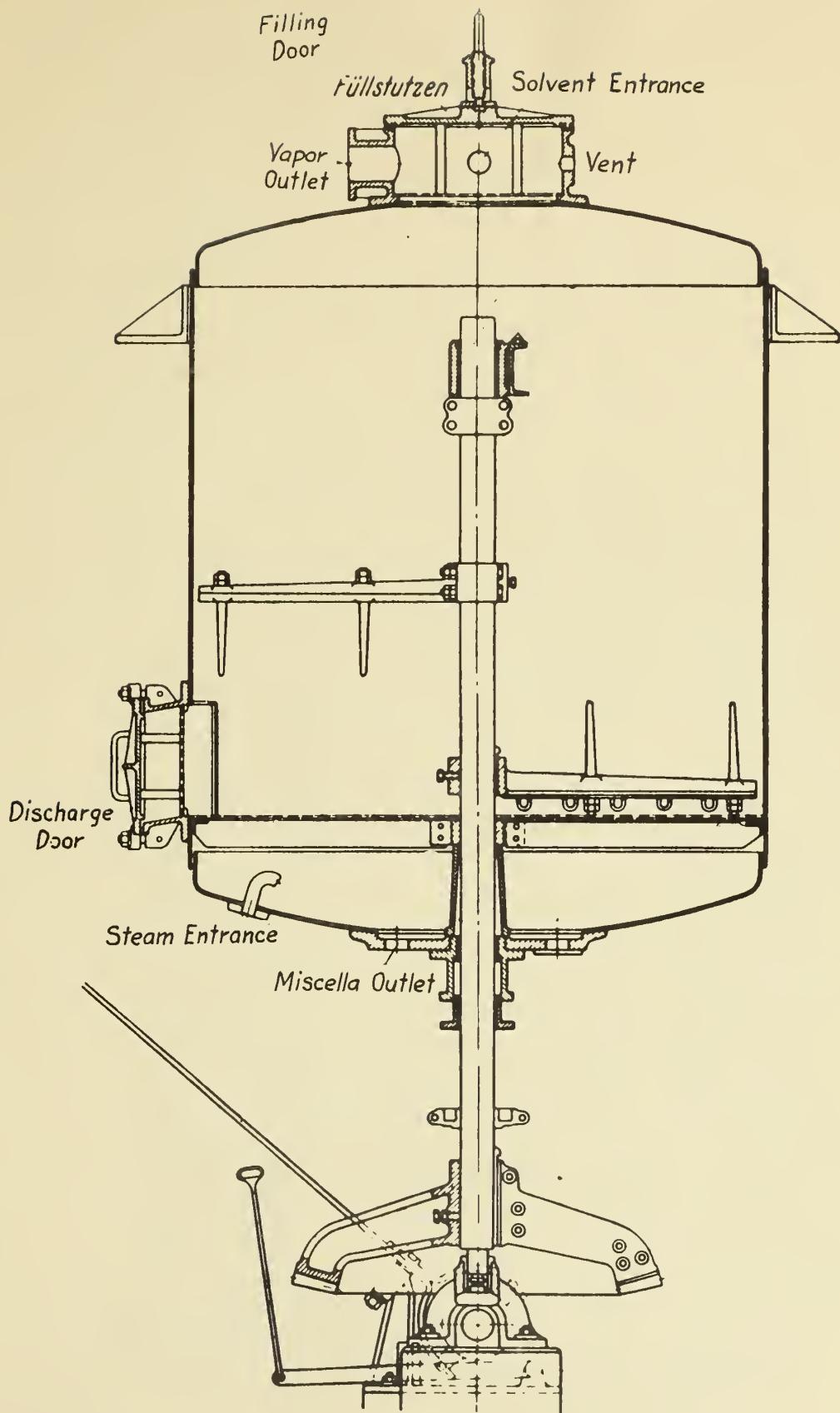


Figure 1—Batch extraction kettle of the type used in batteries for multiple operation.

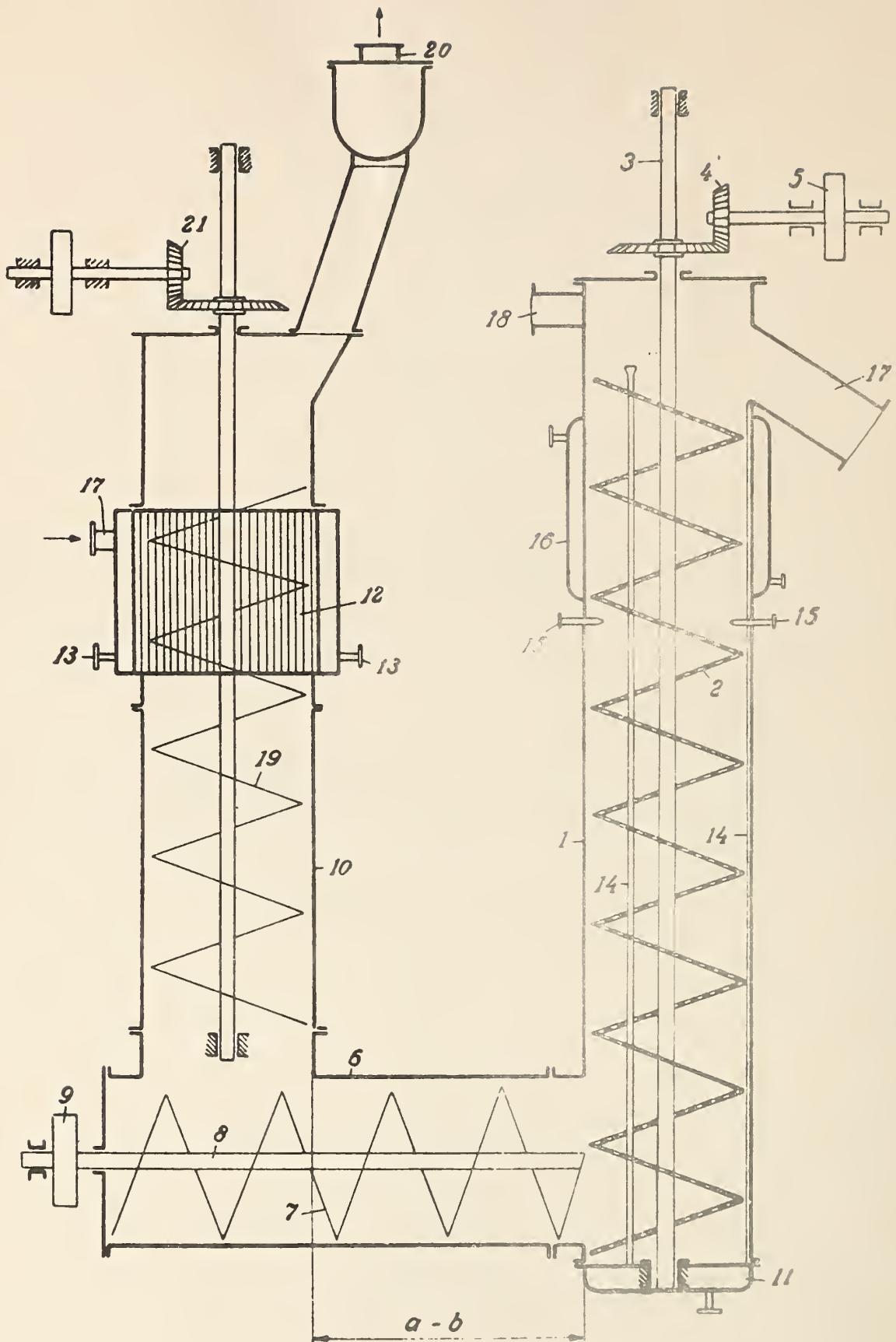


Figure 2—The Hildebrandt extractor.

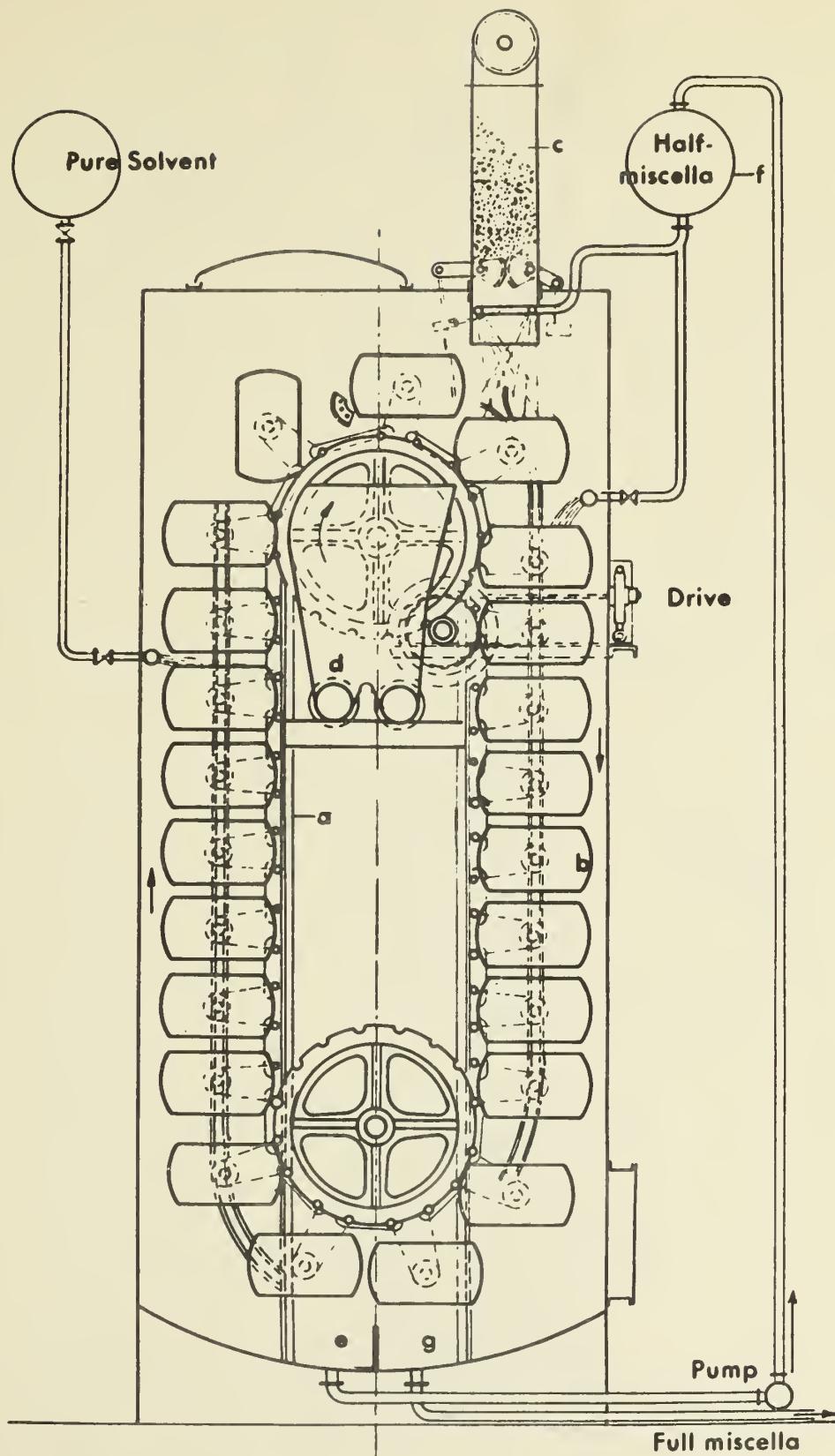


Figure 3—The Böllmann or Hansa-Mühle extractor.

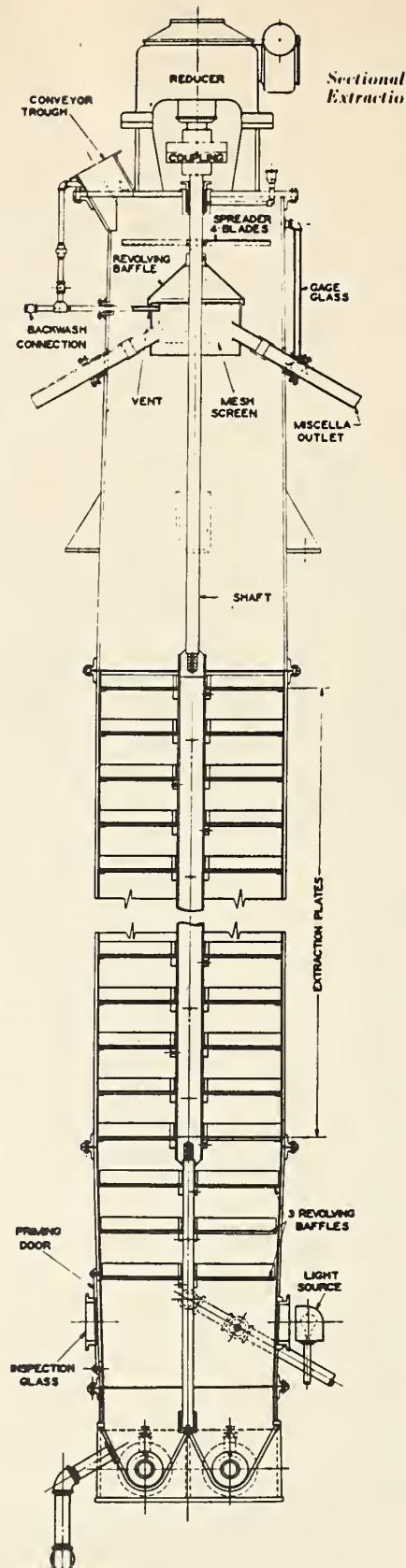


Figure 4—The Bonotto extractor.

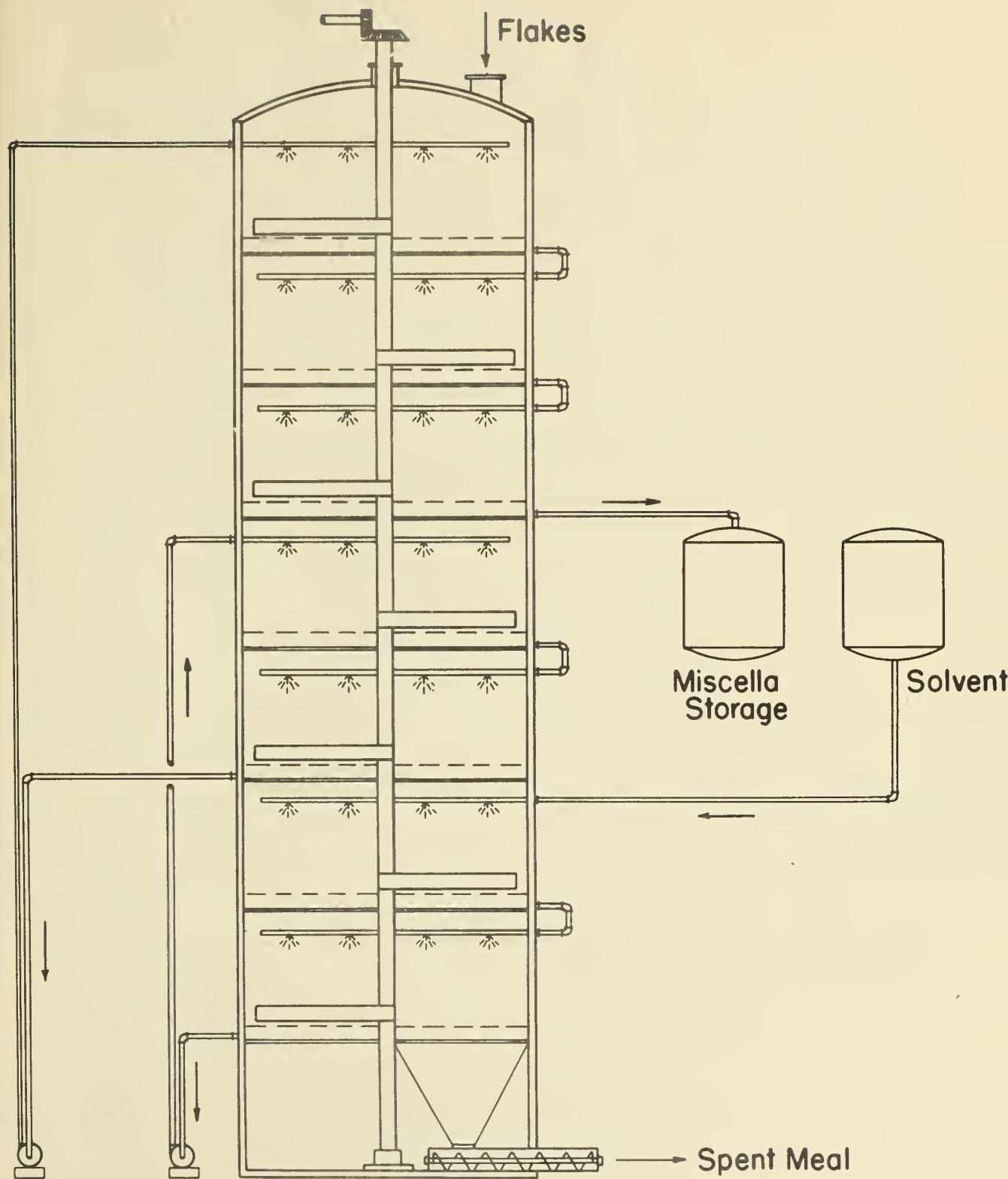


Figure 5—The Tyca extractor.

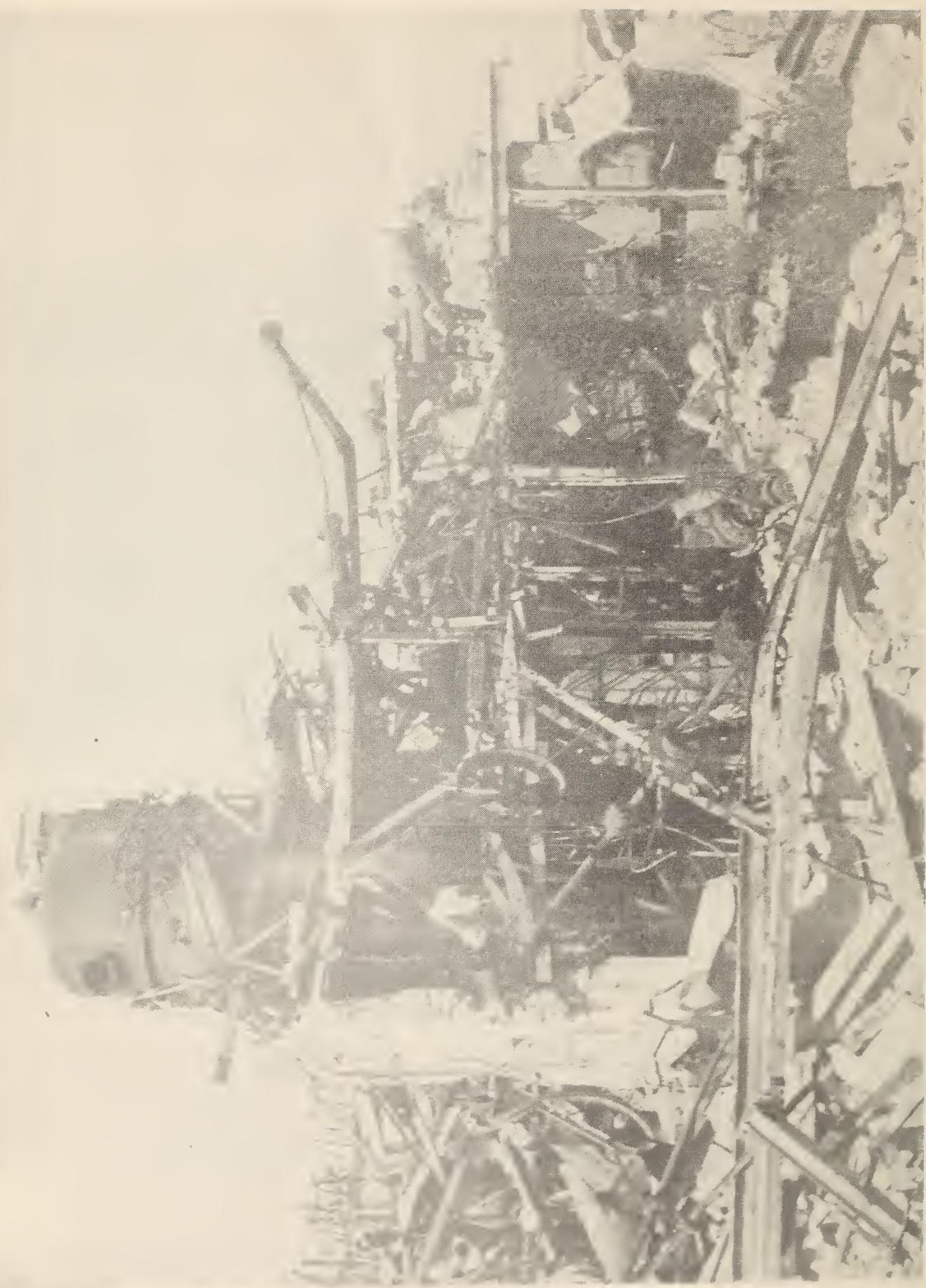


Figure 6—Remains of a Tyca extractor in a German city.

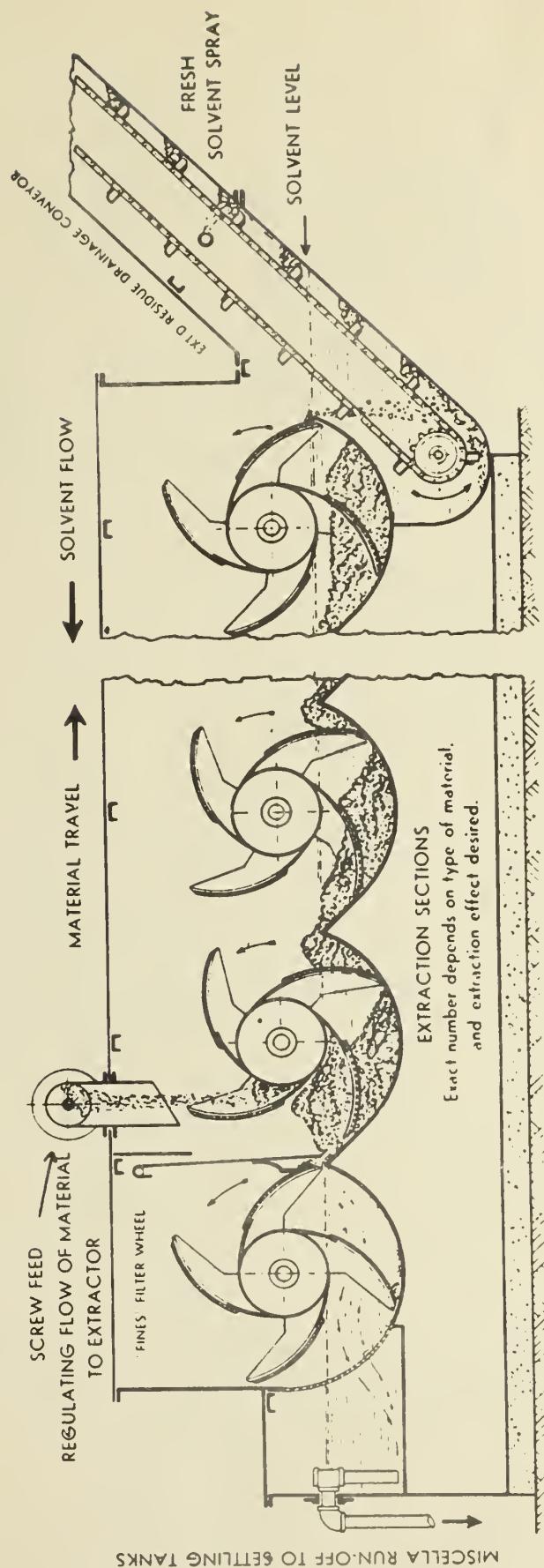


Figure 7 — The Kennedy extractor.

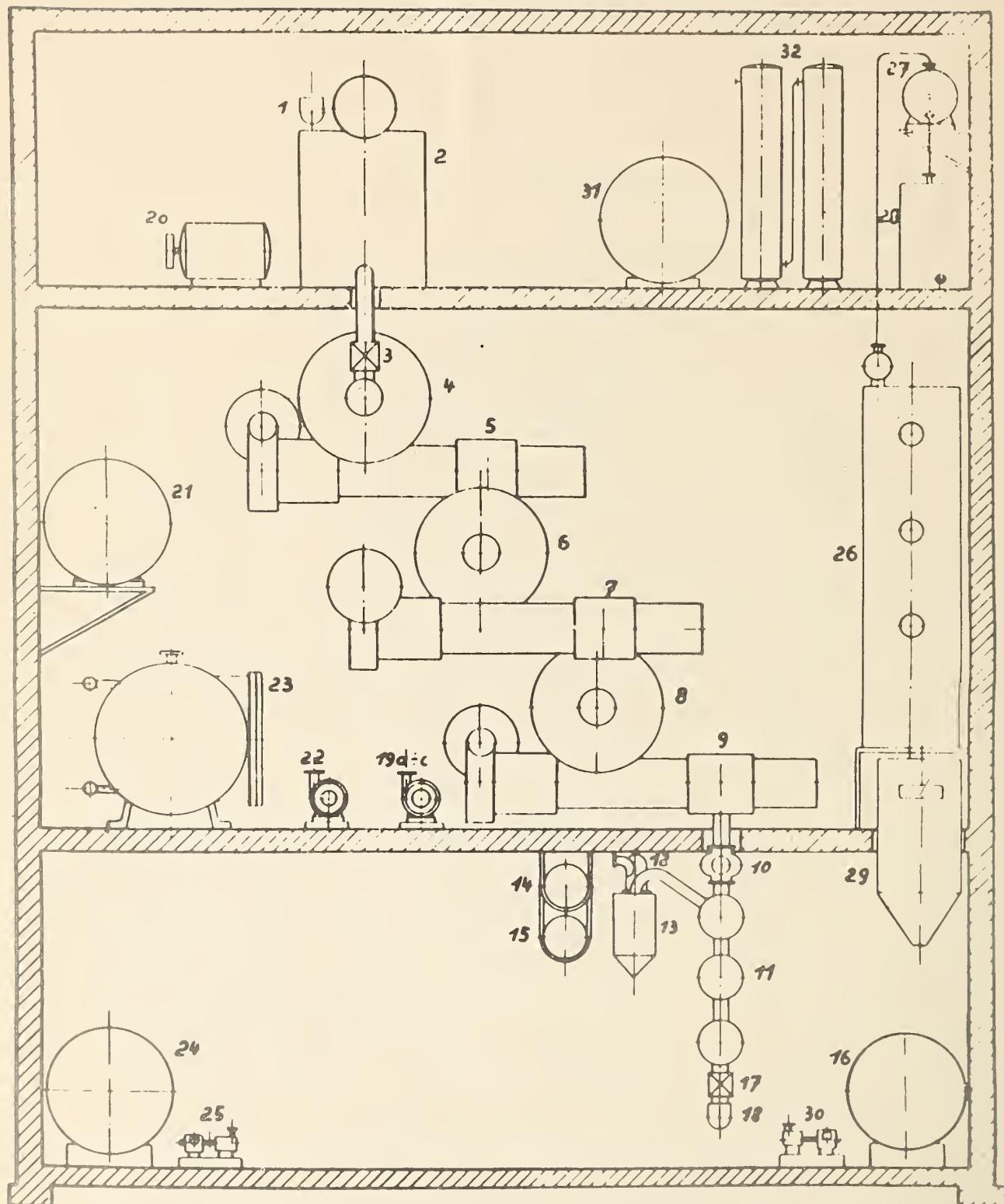


Figure 8—The Fauth system of extraction.

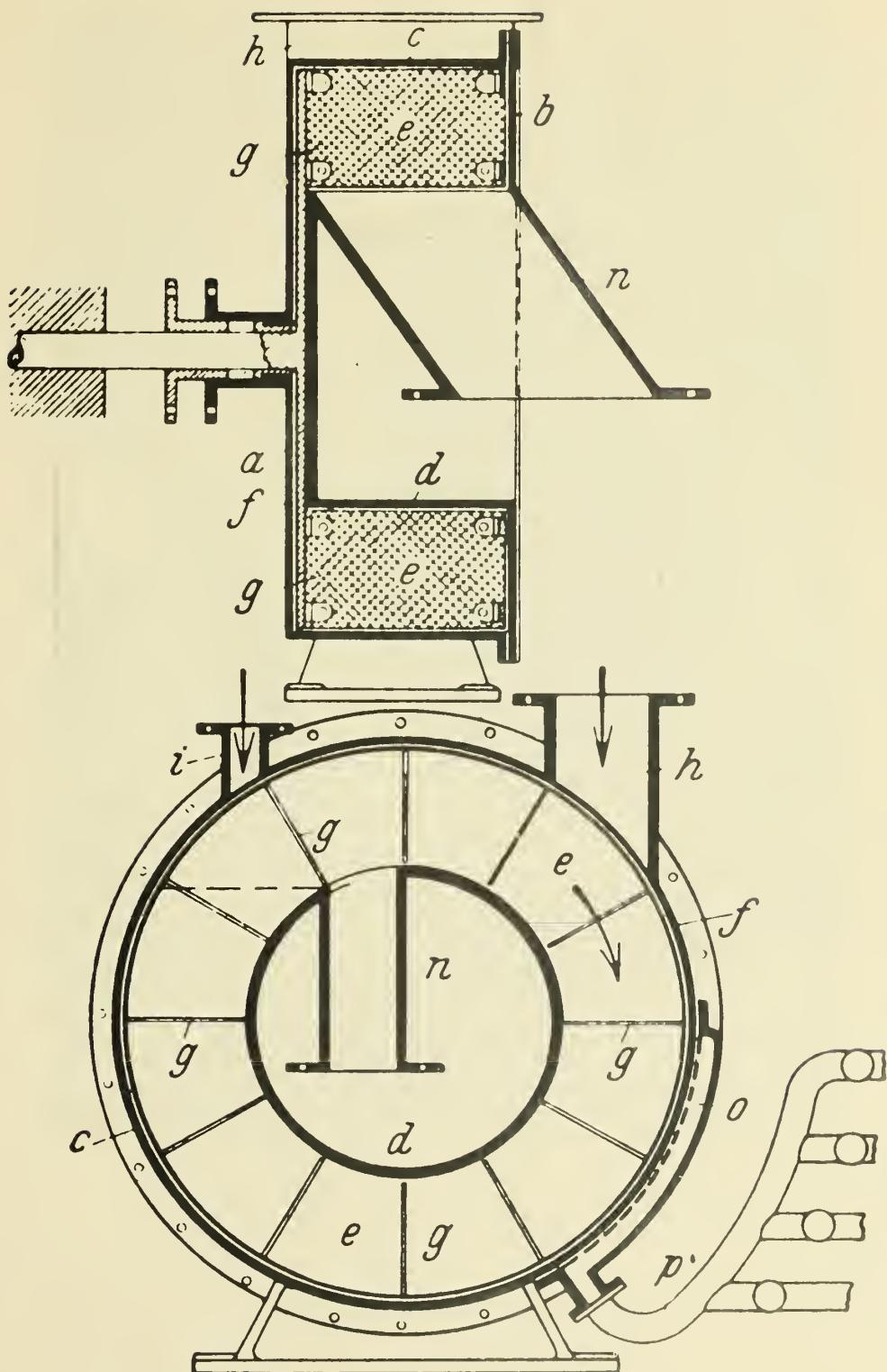


Figure 9—Extractor used in the MIAG system.

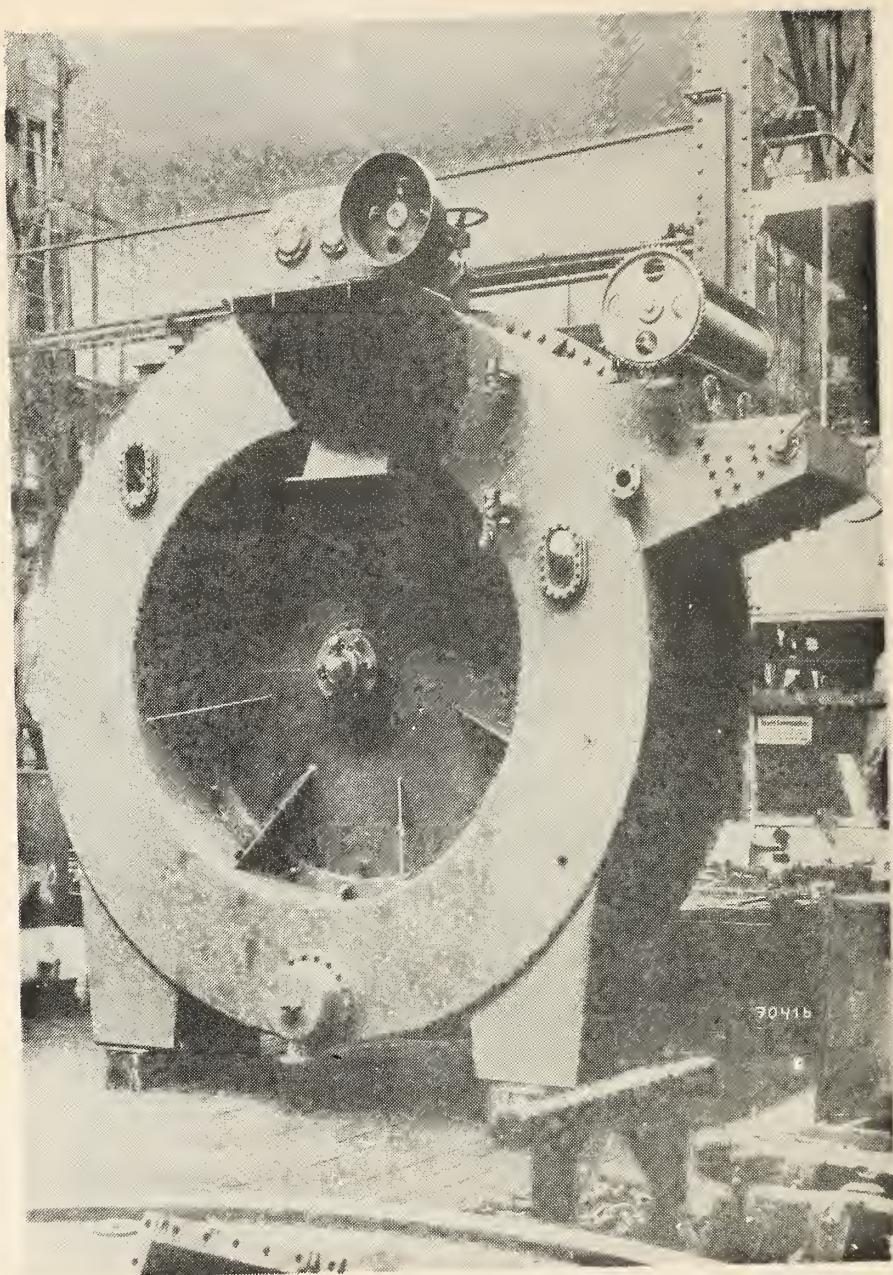


Figure 10—MIAG extractor prior to installation.

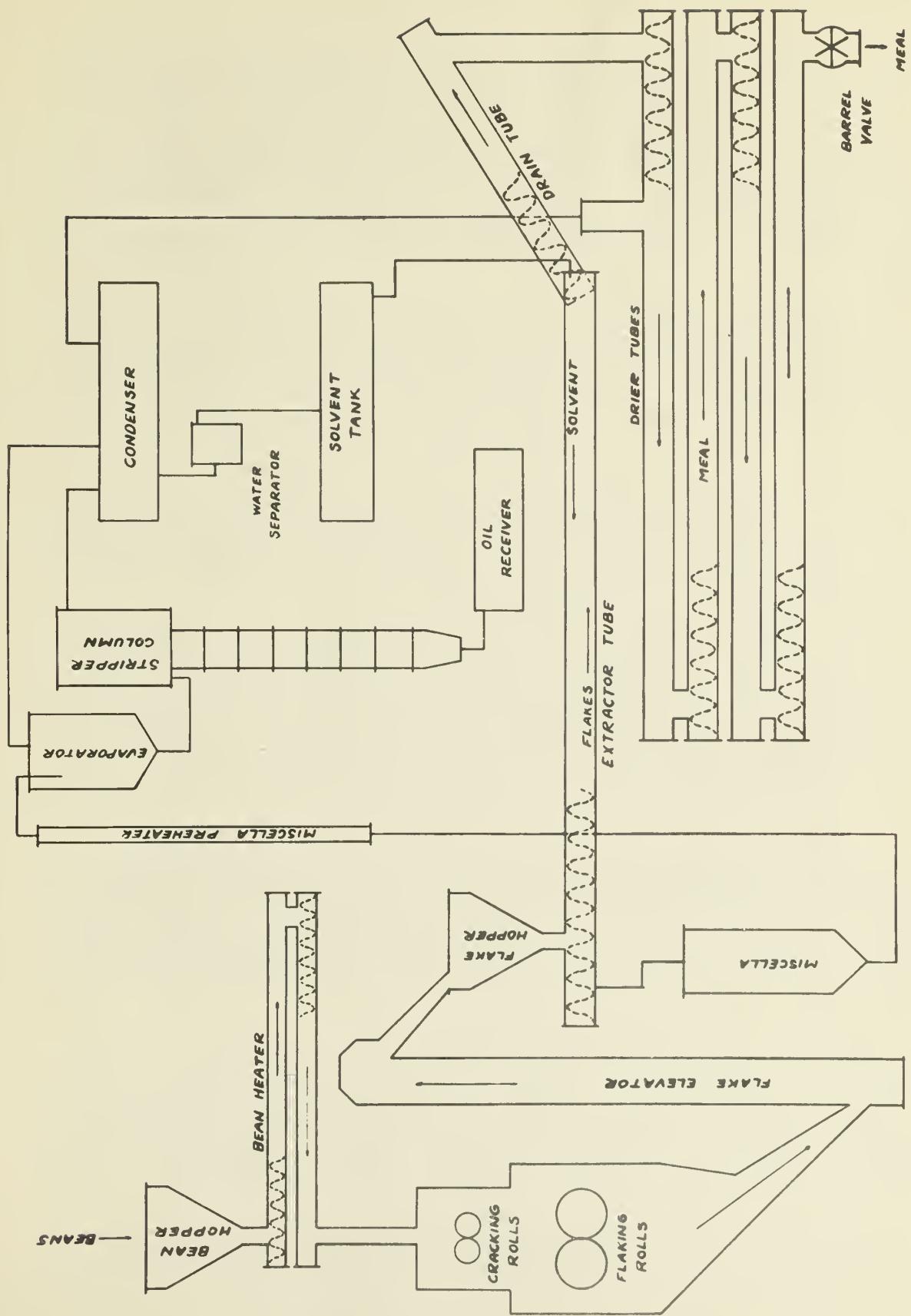


Figure 11—The Detrex system of extraction.

SOLVENTS

The preferred solvent in most oilseed mills is a paraffin hydrocarbon comprised mainly of *n*-hexane, boiling at approximately 150° F. Some German mills used higher-boiling paraffinic solvents before the war, and during the war they were forced to use synthetic hydrocarbons having a relatively wide boiling range and other undesirable properties. Paraffins of the proper volatility have been found, in general, to be the most efficient solvents for removing the oil, along with a minimum of non-oil materials, and subsequently lending themselves to easy separation from the crude oil by distillation. They are extremely flammable, however, and can be used safely only by observing strict precautions against leakage and ignition of vapors which might escape.

Considerable thought has been given to the use of non-flammable solvents, principally the chlorinated hydrocarbons. Trichloroethylene is now used in several very small extractors for processing soybeans, and both this solvent and its homologues are used for extracting a variety of seeds that are processed on a relatively small scale. One English mill employed "tri" in a large extraction plant during the war to minimize the danger of fires during air raids. The chief advantage of trichloroethylene and similar solvents, of course, is their non-flammability and the fact that they can therefore be used on a small scale where it is frequently impractical to observe elaborate precautions against fire. They possess disadvantages, however, including a relatively high cost and a tendency to dissolve non-glyceride materials. If not removed from the oil very thoroughly, these solvents are said to interfere with certain operations in the refining process.

Ethyl alcohol has received consideration as a solvent and, indeed, has been used for some time in one Manchurian soybean mill. It yields a meal having considerably better flavor and color than does extraction with hexane, but its use entails difficulties either in avoiding dilution of the solvent by water or in rectifying the alcohol after it has been diluted in the process. It appears highly probable that in the not very distant future this solvent will be used commercially on at least a small scale for processing soybeans in the United States.

SOLVENT RECOVERY

Extraction systems have become known by the respective mechanical methods used for providing contact between the solvent and the solids. The practices followed in removing the solvent from the products, however, are of greater importance to the quality of the products, particularly the oil, since the solvent must be evaporated from the oil as rapidly as possible at the lowest practicable temperature. The loss of solvent in the entire process of extraction and evaporation is usually a few tenths of 1 percent of the weight of oilseeds processed.

The miscella is ordinarily processed in two or more stages, the first of which is known as pre-evaporation and consists in the removal of about 95 percent of the solvent in evaporators which are usually similar to one of the standard types used in the other chemical processing industries. The commonest are those having vertical tubes, with natural circulation provided through a return leg, and those employing horizontal bundles or coils of steam tubes immersed in the boiling liquid.

Pre-evaporation reduces the concentration of solvent in the oil to a percentage below which the increase in boiling point of the mixture is likely to result in scorching the oil if the operation should be continued. The concentrated miscella is then scrubbed with direct steam in a vertical column containing packing, bubble caps, baffles, or vertical tubes so arranged that the oil flows downward in thin films while being scrubbed countercurrently with the steam.

The spent meal produced in batch extraction is allowed to drain, and live steam is then blown through the mass while a stirrer mixes it thoroughly. In continuous extractors, the marc also drains, and it is then passed through steam-jacketed screw conveyors or a similar drying apparatus. Direct steam is employed in the final stages to assist in carrying away the last traces of solvent.

The vapors from the solvent removal equipment are recovered in condensers, usually of the surface type, and it is necessary to use separate ones for those vapors which contain steam and those which do not. The condensate from the latter is returned directly to the solvent storage tanks for reuse in the extractor, but the condensate containing water must pass through a separator. It is here that extreme difficulty is encountered if the original oilseeds were improperly prepared or if they tend to disintegrate into dust after the oil is removed. Any fine powder in the spent marc is blown into the condensers by the stream of vapor evolved by the residual solids, and the finely powdered meal produces an extremely stable emulsion between the water and the solvent. Although many methods have been devised for minimizing the extent to which these emulsions form, it is practically impossible to prevent their occurrence unless the original seed is properly prepared.

APPLICATIONS

The manner in which solvent extraction is applied to the processing of oilseeds differs in various parts of the world. Until rather recently, Germany was the recognized leader in the technology of extraction, and the methods employed in that country are therefore worthy of study. German operators process soybeans almost entirely by solvent extraction, either in multiple-contact batch or in continuous extractors, but in the United States only about 30 percent of the soybeans processed are extracted. Nearly all of the remainder are pressed in expellers and screw presses. In German mills, peanuts, rapeseed, and other seeds containing high percentages of oil are forepressed and then extracted. This practice is also followed in the United States both on castor beans, which are processed first in cage presses and then in rotary batch extractors, and on wet-milled corn germs which, in some cases, are forepressed in expellers and then extracted in either batch or continuous equipment. In Germany, however, the forepressing is often performed two or three times before extraction, instead of once as in this country.

Copra and palm kernels are ordinarily forepressed in expellers, both in the United States and in Europe. It is customary to "finish" these materials in cage presses, but some European mills have utilized solvent extraction successfully. Sunflowers are processed in a number of ways, but the most common methods are (1) to dehull, forepress in expellers, and extract with solvents and (2) to dehull partly and then extract.

The methods used for processing oilseeds are practically innumerable, as can be seen readily by considering the preceding examples. Solvent extraction is only one of several processes available to the oil miller, but in its modern form it is the most efficient yet developed. Extraction suffers limitations, however, as do all the other processes, and it can be applied satisfactorily only if its limiting factors are thoroughly understood. Although extraction has proved highly successful in the soybean industry, its use for processing linseed, cottonseed, peanuts, and other oilseeds under the conditions peculiar to the United States still presents many unsolved problems. These undoubtedly will be solved, some of them the hard way, for solvent extraction in this country appears destined to expand not only in the oilseed industries where it is now used but also in new fields where other methods have been used exclusively in the past.

